

Skylab 3600 groove/mm replica grating with a scandium–silicon multilayer coating and high normal-incidence efficiency at 38-nm wavelength

John F. Seely, Yu. A. Uspenskii, Yu. P. Pershin, V. V. Kondratenko, and
A. V. Vinogradov

A Sc–Si multilayer coating was applied to a replica of the 3600 groove/mm grating, developed for the SO82A spectroheliograph that flew on the Skylab mission, for the purpose of enhancing the normal-incidence efficiency in the extreme-ultraviolet region. The efficiency, measured at an angle of incidence of 6° with synchrotron radiation, had a maximum value of 7.2% at a wavelength of 38 nm and was a factor of 3 higher than the efficiency of the gold-coated Skylab grating. The measured efficiency of the Sc–Si grating was in good agreement with the efficiency calculated by use of the modified integral method.

OCIS codes: 050.1950, 310.6860, 340.7470.

1. Introduction

Multilayer coated gratings have been developed that have relatively high normal-incidence efficiencies in the extreme-ultraviolet (EUV) wavelength region. Owing to the good performance and stability of Mo–Si multilayers, this type of multilayer was adopted for the first spectrometer planned for a satellite mission using a multilayer grating, the Extreme Ultraviolet Imaging Spectrometer (EIS) on the Solar-B mission.^{1,2} The EIS grating has two Mo–Si coatings that cover the 18–20- and 25–28-nm wavelength ranges. Multilayer gratings with MoRu–Be coatings were recently developed for the 11.5-nm region, and computational modeling of the grating performance indicated that enhanced normal-incidence grating efficiencies can be achieved at shorter wavelengths by use of blazed holographic grating substrates with low microroughness and suitable multilayer coatings.³

The increasing attenuation coefficients of most materials in the EUV region, at wavelengths longer than approximately 30 nm, adversely affect the performance of multilayer coatings. However, the recent

development of Sc–Si multilayer coatings with normal-incidence reflectances exceeding 30% in the 36–48-nm range (Ref. 4) motivated an attempt to demonstrate enhanced grating efficiency near 40 nm. Scandium and the other nearby transition metals have relatively high transmittance at wavelengths longer than the attenuation edge of 3p–3d type transitions.

The 40–44-nm region of the solar spectrum, shown in Fig. 1, has a number of emission lines that span a wide range of temperatures and are of diagnostic importance.⁵ This wavelength region was covered by the gold-coated 3600 groove/mm grating used in the SO82A spectroheliograph that flew on the Skylab mission.⁶ This instrument recorded spectrally dispersed images of the Sun on photographic film. The exposure time of the spectral image shown in Fig. 1 was 8.5 s.⁷ To increase significantly the temporal cadence of the images for the study of transient solar phenomena, it would be necessary to increase the efficiency of the grating. Thus, for this study, a replica of the Skylab 3600 groove/mm grating was chosen as the grating substrate so that direct comparisons could be made with previous efficiency measurements of similar replicas with Mo–Si multilayer and gold coatings.⁸

2. Scandium–Silicon Multilayer Coating

A number of Sc–Si multilayer test coatings were deposited onto Si wafer substrates. A thin W barrier layer was deposited between the Sc and the Si layers to prevent interdiffusion at the Sc–Si interfaces and to improve the stability of the multilayers.⁹ The reflectances of the test mirrors were

J. F. Seely (john.seely@nrl.navy.mil) is with the Naval Research Laboratory, Space Science Division, Code 7674, Washington D.C. 20375. A. Vinogradov and Yu. A. Uspenskii are with the X-Ray Optics Group, Lebedev Physical Institute, 117924 Moscow, Russia. Yu. P. Pershin and V. V. Kondratenko are with the Kharkov State Polytechnic University, Kharkov 61002, Ukraine.

Received 20 July 2001; revised manuscript received 7 November 2001.

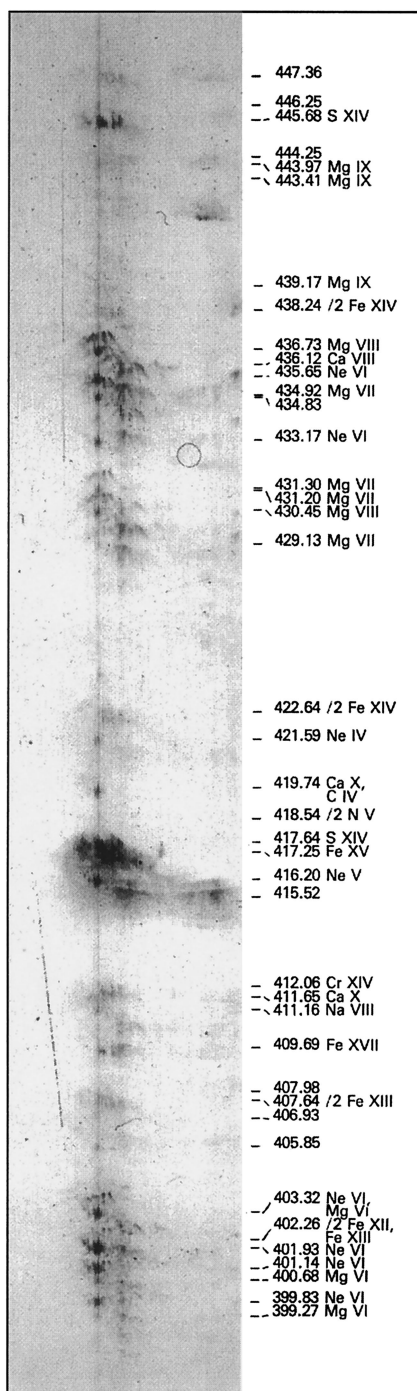


Fig. 1. Solar spectrum in the 39–45-nm wavelength range from the 2 December 1973 impulsive EUV event recorded by the NRL SO82A spectroheliograph on Skylab.

measured at the Naval Research Laboratory (NRL) beamline X24C at the National Synchrotron Light Source at the Brookhaven National Laboratory.^{10,11} The angle of incidence was 5°, and the incident radiation was 80% polarized with the electric field vector in the plane of incidence. The measured reflectance of a Sc–W–Si–W multilayer is shown by the data points in Fig. 2. The coating had 20 periods with a nominal Sc fractional thickness of 0.45.

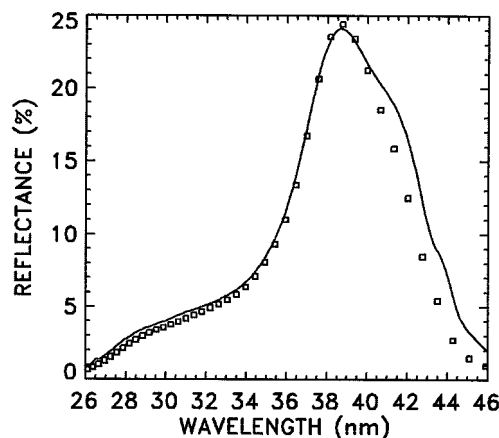


Fig. 2. Measured (data points) and calculated (curve) reflectance of a Sc–Si test mirror.

The thickness of the W layers was 0.8 nm. The top layer was oxidized Si with an assumed thickness of 3 nm as is usually adopted. The peak reflectance of the multilayer shown in Fig. 2 is 25% at a wavelength of 39 nm.

Initial attempts to model the reflectance of the Sc–Si multilayer indicated that the tabulated (Ref. 12) optical constants for Sc are inaccurate in the 35–45-nm wavelength range. Thus it was necessary to determine experimentally the Sc optical constants. This was done by means of depositing one Sc–Si bilayer onto each of six Si photodiode sensors and measuring the transmittances. The Si layer thickness (5 nm) was the same on each photodiode, and the Sc thickness varied from 7.5 to 130 nm. The Sc optical constants were determined in the 18–68-nm wavelength range.¹³

The reflectances of the Sc–Si test mirrors were calculated with the newly determined Sc optical constants and the Si and W optical constants from Ref. 14. The interfaces were assumed to be discrete with no interdiffusion or microroughness. The only free parameter in the calculation was the period thickness, which was adjusted so that the wavelength of the peak calculated reflectance agreed with the measurements. The resulting calculated reflectance is shown by the solid curve in Fig. 2. The calculated and the measured reflectances are in excellent agreement except on the long wavelength side of the reflectance profile. The inferred period thickness is 20.3 nm.

3. Measured Grating Efficiency

A similar Sc–Si multilayer coating with W barrier layers was applied to a replica of the Skylab 3600 groove/mm grating. The efficiency was measured at the NRL beamline X24C by means of scanning the detector in angle through the grating orders. The efficiencies were measured at two angles of incidence, 6° and 15.5°, relative to the normal to the grating surface. The incident radiation was approximately 80% polarized with the electric field vector perpen-

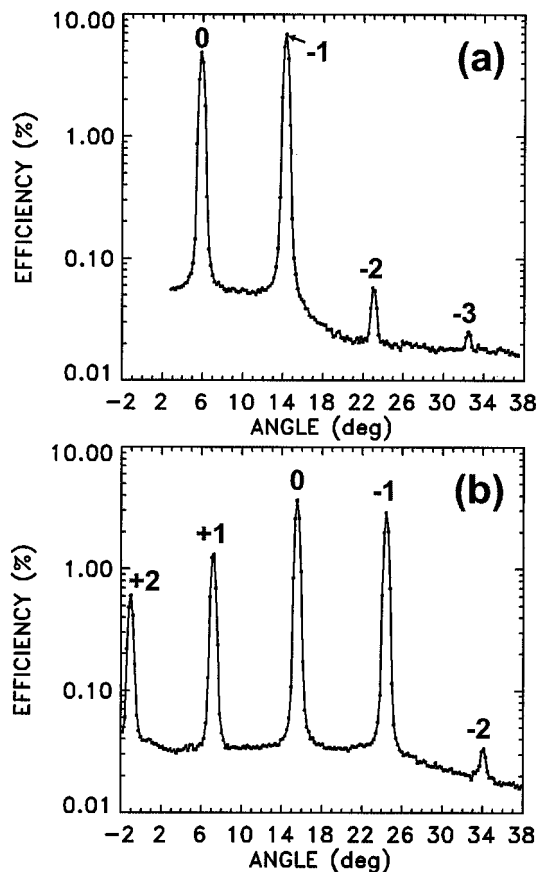


Fig. 3. Sc-Si grating efficiencies measured at a wavelength of 40 nm and at angles of incidence of (a) 6° and (b) 15.5°.

pendicular to the grating's grooves. The grating was oriented so that the groove facets with the larger facet angles faced the incident radiation beam. In this orientation the outside first diffraction order, with a diffraction angle larger than the 0 order, had the highest efficiency. The efficiencies measured at an incident wavelength of 40 nm are shown in Fig. 3, where negative and positive numbers identify the outside and the inside orders, respectively. The efficiencies measured at an incident wavelength of 19 nm, near the peak of the second constructive interference (Bragg) order of the Sc-Si multilayer coating, are shown in Fig. 4. As discussed in Section 4, the efficiencies at the shorter wavelengths are small, owing to the low groove efficiency of the grating substrate at the shorter wavelengths and, in addition, to the low multilayer reflectance in the second Bragg order.

The peak efficiencies in the -1 and the 0 grating diffraction orders, measured as functions of the incident wavelength at an angle of incidence of 6°, are shown by the data points in Fig. 5. The peak -1 efficiency is 7.2% at a wavelength of 38 nm. This is a factor of 3 higher than the efficiency of the gold-coated Skylab grating.^{6,8} The curves in Fig. 5 are the calculated efficiencies that are discussed in Section 4.

The period thickness of the grating's Sc-Si mul-

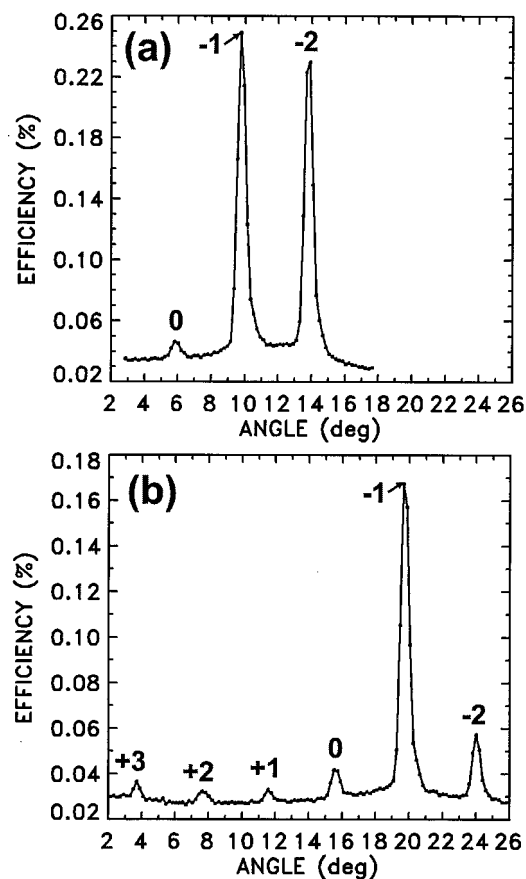


Fig. 4. Sc-Si grating efficiencies measured at a wavelength of 19 nm and at angles of incidence of (a) 6° and (b) 15.5°.

tilayer coating was determined by measurement of the 0-order efficiency over a wide wavelength range. This was done by means of positioning the detector at the 0-order angle, which does not change with wavelength, and scanning the incident wavelength at a fixed angle of incidence of 6°. The measured

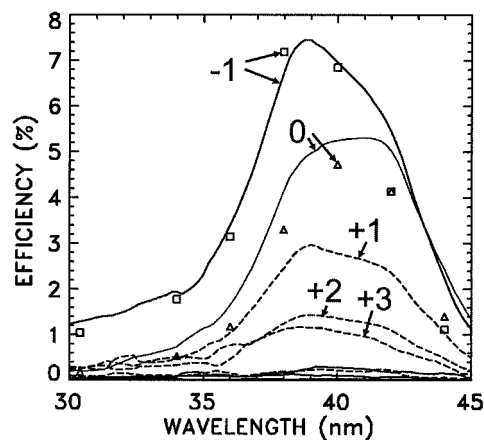


Fig. 5. Data points are the measured peak efficiencies of the Sc-Si grating in the -1 and the 0 orders for an angle of incidence of 6°. The curves are the calculated efficiencies at an angle of 6°.

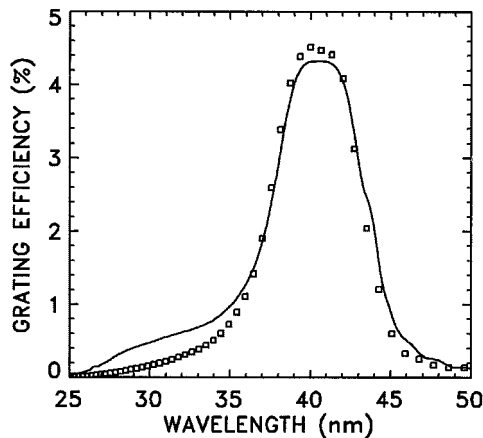


Fig. 6. Data points are the measured 0-order efficiency of the Sc-Si grating at an angle of incidence of 6° . The curve is the calculated reflectance of a Sc-Si multilayer coating at 6° incidence and multiplied by 0.21, which represents the 0-order groove efficiency.

0-order efficiency is shown by the data points in Fig. 6.

The curve in Fig. 6 is the calculated reflectance of the Sc-Si coating. The reflectance was calculated as for the Sc-Si test coatings on the flat Si wafers, except that the reflectivity at each layer interface was multiplied by a Debye-Waller factor to simulate the 1.6-nm microroughness of the grating substrate.⁸ The period thickness was varied so that the wavelength of the peak of the calculated reflectance profile agreed with the peak of the measured 0-order efficiency. In addition, the entire reflectance profile was scaled down by a factor of 0.21, which represents the grating's groove efficiency in the 0 order. As shown in Fig. 6, with this scaling, the calculated reflectance is in good overall agreement with the measurements. The inferred multilayer period thickness is 20.9 nm.

4. Calculated Grating Efficiency

The efficiency of the Sc-Si multilayer coated grating was calculated with the computer program PCGRATE developed by Goray.¹⁵⁻¹⁸ This program implements the modified integral method to solve the boundary value problem of electromagnetic radiation incident on a diffraction grating with a multilayer coating. The program accounts for the polarization of the incident radiation, the grating's groove profile as determined by atomic force microscopy, and the optical properties of the layers. The program has been used for accurately modeling, in the 11–50-nm wavelength range, the efficiencies of blazed gratings of three types: uncoated holographic master grating, uncoated replica of the master grating, and replica gratings with MoRu-Be multilayer coatings.^{19,20} Although the program accounts for the microroughness of the groove profile of the grating substrate, it does not currently account for the effect of layer microroughness on the reflectivity. Thus the calculated efficiencies were multiplied by the Debye-

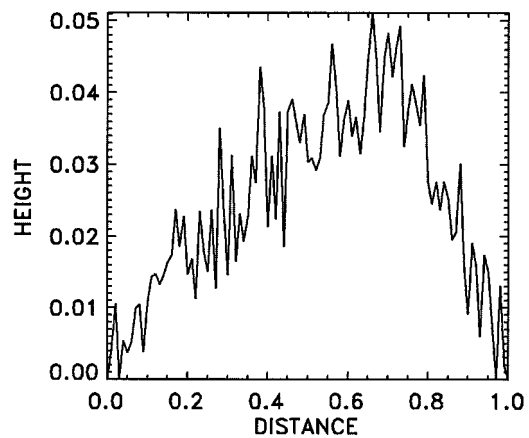


Fig. 7. Groove profile used for the calculation of the Sc-Si grating efficiency. The groove dimensions are normalized to the groove period (278 nm)

Waller factor $\exp[-(2\pi m\sigma/d)^2]$ to account for layer microroughness.²¹ This is only an approximation for a proper detailed accounting of layer microroughness that is planned for implementation in the program.

The groove profile, derived from the atomic force microscopy images of replicas of the Skylab grating,⁸ is shown in Fig. 7. The nominal angles of the left and right facing facets are 3.1° and 7.3° , respectively. The groove profile has 1.6-nm rms microroughness with respect to the nominal groove profile.

As shown by the curves in Figs. 5 and 8(a), the efficiencies calculated at an angle of incidence of 6° are in good agreement with the measured -1 and 0 peak efficiencies [shown by the data points in Figs. 5 and 8(a)] in both the first and the second Bragg orders of the multilayer coating near wavelengths of 38 and 19 nm, respectively. The calculated groove efficiencies are shown in Fig. 8(b). The crossings of the -1 and the 0 orders near 43 nm and the -1 and -2 orders near 18 nm are consistent with the present measurements and with the previous measurements of a Skylab replica grating with a Mo-Si multilayer coating in the 11–34-nm wavelength range.⁸

The efficiency of a multilayer coated grating is essentially the product of the groove efficiency of the grating substrate and the reflectance of the multilayer coating. The groove profile determines the relative efficiencies in the various diffraction orders, and the multilayer's period thickness determines the wavelength of peak reflectance. The groove profile and the period thickness are usually optimized so that the multilayer grating has peak efficiency in a diffraction order and wavelength region of interest. In the case of a blazed grating substrate, the blaze angle is selected to give maximal efficiency in the desired on-blaze order and low efficiency in the other diffracted orders and in the 0 order.²² It is apparent from Fig. 8(b) that the Skylab replica grating does not have optimal groove profile for the 38-nm wavelength region. The 3.1° blaze angle results in peak -1 -order efficiency near 30 nm, the design wavelength of

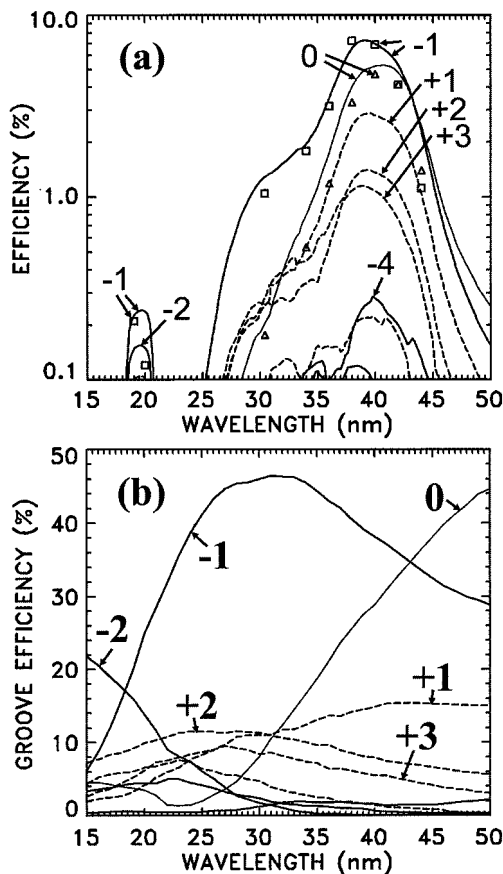


Fig. 8. (a) Comparison of the measured (data points) Sc-Si grating efficiency in the -1 and the 0 orders and the calculated efficiencies (curves) at an angle of incidence of 6° . (b) The calculated groove efficiency at 6° incidence.

the Skylab grating.⁶ Thus at 38 nm, the -1 -order efficiency is decreasing and the 0 -order efficiency is comparable and is increasing. The Skylab replica grating was selected from this study because of the previous detailed characterizations of replicas with gold and Mo-Si multilayer coatings.⁸

Shown in Fig. 9(a) is the calculated groove efficiency of a 3600 groove/mm grating with a blaze angle of 4.75° , which corresponds to a blaze wavelength of 40 nm. The opposite groove facets have an angle of 12° . The peak efficiency in the -1 order, calculated at an angle of incidence of 6° , is 54% at a wavelength of 40 nm. The efficiencies in the other orders are much lower as shown in Fig. 9(a). The -2 -order efficiency is high in the 20–25-nm range, and a grating with these parameters was used in second order to record solar spectra on the Solar Extreme Ultraviolet Research Telescope and Spectrograph (SERTS) rocket mission.^{23–25} The efficiency of the grating with a Sc-Si multilayer coating optimized for a wavelength of 42 nm is shown in Fig. 9(b). The peak efficiency is 15%, and the efficiency exceeds 12% in the 39.4–43.4-nm wavelength range. A grating of this type would be useful for the spectroscopic study of the solar spectrum in the 39–44-nm wave-

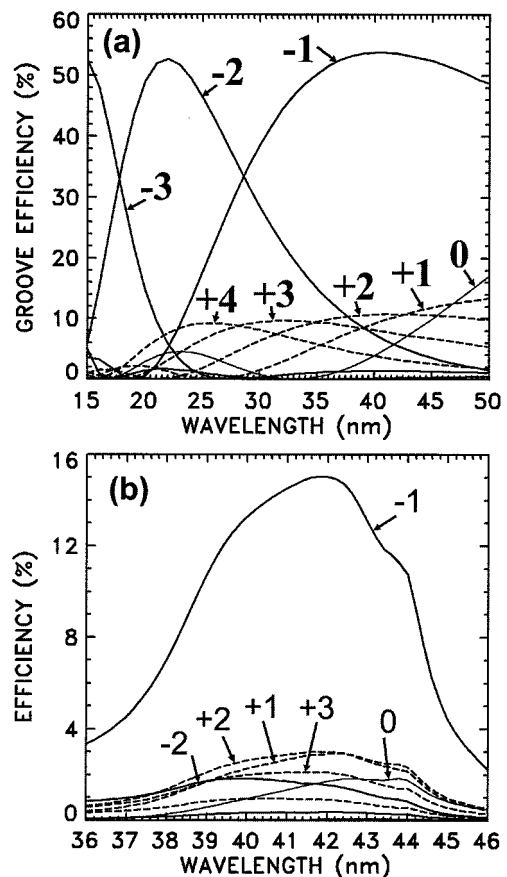


Fig. 9. (a) Calculated groove efficiency of the optimized grating substrate. (b) The calculated efficiency of the optimized Sc-Si grating.

length range (see Fig. 1). The peak grating efficiency is a factor of 5 higher than the gold-coated Skylab grating, and the comparative spectral image exposure cadence would be 5 times faster. By comparison, the peak efficiency of the EIS (Solar-B mission) Mo-Si multilayer grating is 9% in the 26-nm wavelength region.²

5. Conclusion

The Sc-Si multilayer coating greatly increased the normal-incidence efficiency of a replica of the Skylab 3600 groove/mm grating in the 40-nm wavelength region, which is important for the study of the solar corona with high-resolution spectroscopic diagnostic techniques. The peak efficiency was 7.2%, which was lower than optimum because of the small blaze wavelength (30 nm) of the grating substrate. Modeling indicates that an optimized grating with a Sc-Si multilayer coating would have a peak efficiency of 15% at a wavelength of 42 nm and an efficiency exceeding 12% over a 4-nm-wide wavelength range. In addition, the newly determined optical constants indicate that Sc may not be the best multilayer material for the 40–42-nm wavelength region. It was found that the experimentally determined Sc atten-

uation coefficient was higher than expected, owing to absorption by 3p–3d type transitions below a wavelength of approximately 43 nm. An experimental study of the optical constants of Ti indicates that the 3p–3d absorption threshold is at a wavelength of approximately 35 nm, and Ti has lower attenuation and is expected to have better multilayer performance than Sc in the 40-nm region. Thus there is the potential that the peak normal-incidence efficiency of an optimized Ti–Si multilayer grating may be considerably higher than 15% in the 40-nm wavelength region.

The authors are indebted to I. A. Artioukov, R. M. Fechtchenko, and N. L. Popov for fruitful discussions and cooperation. This research was supported by NASA grant S-13617 and by Civilian Research and Development Foundation grants NN RPO-882 and RPI-2267. Part of the research was performed at the National Synchrotron Light Source which is supported by the U.S. Department of Energy.

References and Note

1. J. L. Culhane, C. M. Korendyke, T. Watanabe, and G. A. Doschek, "An extreme ultraviolet imaging spectrometer designed for the Japanese Solar-B satellite," in *Instrumentation for UV/EUV Astronomy and Solar Missions*, S. Fineschi, C. Korendyke, O. Siegmund, and B. Woodgate, eds., Proc. SPIE **4139**, 294–312 (2000).
2. J. F. Seely, "Multilayer grating for the extreme ultraviolet spectrometer (EIS)," in *X-Ray Optics, Instruments, and Missions IV*, R. Hoover and A. Walker, eds., Proc. SPIE **4138**, 174–181 (2000).
3. C. Montcalm, S. Bajt, and J. F. Seely, "MoRu–Be multilayer-coated grating with 10.4% normal-incidence efficiency near the 11.4-nm wavelength," Opt. Lett. **26**, 125–127 (2001).
4. Yu. A. Uspenskii, V. E. Levashov, A. V. Vinogradov, A. I. Fedorenko, V. V. Kondratenko, Yu. P. Pershin, E. N. Zubarev, and V. Yu. Fedotov, "High-reflectivity multilayer mirrors for a vacuum-ultraviolet interval of 35–50 nm," Opt. Lett. **23**, 771–773 (1998).
5. U. Feldman, "Elemental abundances in the upper solar atmosphere," Phys. Scr. **46**, 202–220 (1992).
6. R. Tousey, J.-D. F. Bartoe, G. E. Brueckner, and J. D. Purcell, "Extreme ultraviolet spectroheliograph ATM experiment SO82A," Appl. Opt. **16**, 870–878 (1977).
7. U. Feldman, J. D. Purcell, and B. Dohne, *An Atlas of Extreme Ultraviolet Spectroheliograms from 170 to 625 Å* (E. O. Hulburt Center for Space Research, Naval Research Laboratory, Washington, D.C., 1987).
8. J. F. Seely, M. P. Kowalski, W. R. Hunter, T. W. Barbee, R. G. Cruddace, and J. C. Rife, "Normal-incidence efficiencies in the 115–340 Å wavelength region of replicas of the Skylab 3600 line/mm grating with multilayer and gold coatings," Appl. Opt. **34**, 6453–6458 (1995).
9. D. L. Voronov, E. N. Zubarev, V. V. Kondratenko, A. V. Pen'kov, Yu. P. Pershin, A. G. Ponomarenko, A. V. Vinogradov, Yu. A. Uspenskii, and J. F. Seely, "Structure, thermal stability and reflectivity of Sc/Si and Sc/W/Si/W multilayer x-ray mirrors," in *Soft X-Ray Lasers and Applications IV*, E. E. Fill and J. J. G. Rocca, eds., Proc. SPIE **4505** (to be published).
10. J. C. Rife, H. R. Sadeghi, and W. R. Hunter, "Upgrades and recent performance of the grating/crystal monochromator," Rev. Sci. Instrum. **60**, 2064–2067 (1989).
11. W. R. Hunter and J. C. Rife, "An ultrahigh vacuum reflectometer/goniometer for use with synchrotron radiation," Nucl. Instrum. Methods A **246**, 465–468 (1986).
12. B. L. Henke, E. M. Gullikson, and J. C. Davis, "X-ray interactions: photoabsorption, scattering, transmission, and reflection at E=50–30,000 eV, Z=1–92," At. Data Nucl. Data Tables **54**, 181–342 (1993).
13. Yu. A. Uspenskii, J. F. Seely, N. L. Popov, A. V. Vinogradov, Yu. P. Pershin, and V. V. Kondratenko are preparing a manuscript to be called "New method for the determination of EUV optical constants in chemically active materials: application to Sc and Ti."
14. E. D. Palik, *Handbook of Optical Constants of Solids* (Academic, San Diego, Calif., 1985).
15. L. I. Goray, "Numerical analysis for relief gratings working in the soft x-ray and XUV region by the integral equation method," in *X-Ray and UV Detectors*, R. B. Hoover and M. W. Tate, eds., Proc. SPIE **2278**, 168–172 (1994).
16. L. I. Goray, "Rigorous integral method in application to computing diffraction on relief gratings working in wavelength range from microwaves to x-ray," in *Application and Theory of Periodic Structures*, T. Jansson and N. C. Gallagher, eds., Proc. SPIE **2532**, 427–433 (1995).
17. L. I. Goray, "Modified integral method for weak convergence problems of light scattering on relief grating," in *Diffraction and Holographic Technologies for Integrated Photonic Systems*, R. I. Sutherland, D. W. Prather, and I. Cindrich, eds., Proc. SPIE **4291** (to be published).
18. L. I. Goray, "Modified integral method and real electromagnetic properties of echelles," in *Diffraction and Holographic Technologies for Integrated Photonic Systems*, R. I. Sutherland, D. W. Prather, and I. Cindrich, eds., Proc. SPIE **4291** (to be published).
19. J. F. Seely and L. I. Goray, "Normal incidence multilayer gratings for the extreme ultraviolet region: experimental measurements and computational modeling," in *X-Ray Optics, Instruments, and Missions II*, R. B. Hoover and A. B. Walker, eds., Proc. SPIE **3766**, 364–370 (1999).
20. L. I. Goray and J. F. Seely, "Efficiencies of master, replica, and multilayer gratings for the soft x-ray–extreme-ultraviolet range: modeling based on the modified integral method and comparisons with measurements," Appl. Opt. (to be published).
21. E. Spiller, *Soft X-Ray Optics* (SPIE, Bellingham, Wash., 1994), p. 110.
22. J. F. Seely, M. P. Kowalski, W. R. Hunter, J. C. Rife, T. W. Barbee, G. E. Holland, C. N. Boyer, and C. M. Brown, "On-blaze operation of a Mo/Si multilayer-coated concave diffraction grating in the 136–142 Å wavelength region and near normal incidence," Appl. Opt. **32**, 4890–4897 (1993).
23. J. W. Brosius, J. M. Davila, and R. J. Thomas, "Calibration of the SERTS-95 spectrograph from iron line intensity ratios," Astrophys. J. **497**, L113–L116 (1998).
24. R. A. M. Keski-Kuha, R. J. Thomas, J. S. Gum, and C. E. Condor, "Performance of multilayer coated diffraction gratings in the EUV," Appl. Opt. **29**, 4529–4531 (1990).
25. R. J. Thomas, R. A. M. Keski-Kuha, W. M. Neupert, C. E. Condor, and J. S. Gum, "Extreme ultraviolet performance of a multilayer coated high-density toroidal grating," Appl. Opt. **30**, 2245–2251 (1991).